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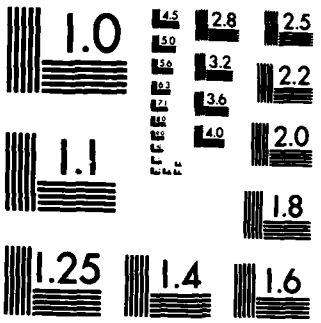

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A double-beam instrument developed in this laboratory has been used to measure the complex indices of refraction of various materials at 245 GHz. We report here the results for crystal quartz, water free fused quartz (Spectrasil WF), silicon, beryllia, boron nitride (grade HP), and a nickel ferrite (Trans-Tech 2-111). We compare our results with the results obtained by other researchers using different techniques as available.

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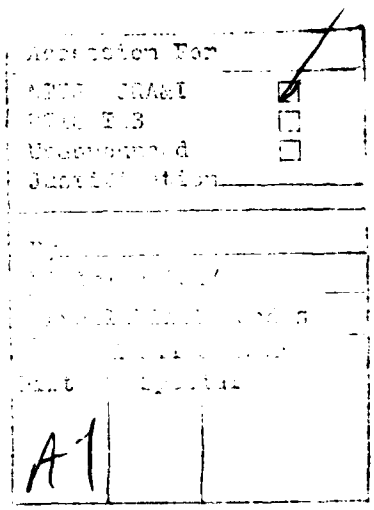
# HIGH PRECISION MATERIALS CHARACTERIZATION IN THE NEAR MILLIMETER WAVE-LENGTH RANGE

FINAL REPORT

Dr. Jyotsna Moy Dutta
Dr. Charles R. Jones

February 7, 1985

U. S. ARMY RESEARCH OFFICE

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## ABSTRACT

A double-beam instrument developed in this laboratory has been used to measure the complex indices of refraction of various materials of 245 GHz. We report here the results for crystal quartz, water free fused quartz (Spectrasil WF), silicon, beryllia, boron nitride (grade HP), and a nickel ferrite (Trans-Tech 2-111). We compare our results with the results obtained by other researchers using different techniques as available.

## I. Introduction

There is a growing interest in potential applications for the near-millimeter-wave (NMMW) spectral region (defined approximately as wavelengths between 0.3 mm and 3 mm). This interest is not limited to laboratory studies of interesting physical phenomena, but includes increasing military and civilian requirements. Rapid advances are being made in the development of sources, detectors, mixers, etc. However, the development of functional systems in the NMMW region is dependent also on the availability of improved components, including windows, attenuators, isolators, modulators, switches, directional couplers, etc. The development of such devices requires accurate data on the dielectric properties of materials in this spectral region. An extensive survey of the literature (1) has revealed a serious shortage of data, particularly in the 100 to 300 GHz region. Disagreement in NMMW measurements by different researchers is not uncommon. This may be due to large measurement uncertainties or to differences between nominally identical samples.

The present work involves the use of a double-beam instrument developed in this laboratory for the accurate measurement of dielectric properties. This instrument utilizes the power and spatial coherence of a laser source and combines the use of "quasi-optical" techniques with the introduction of over-moded dielectric waveguides to limit diffraction spreading of the radiation. The capability of this instrument to provide accurate values for the refractive index and absorption coefficient at 245 GHz has been demonstrated for a variety of low-loss materials (2). We report here on the extension of these measurements to a variety of interesting materials which have low to moderate losses.

## II. Experimental Design

All measurements were performed at a frequency of 245 GHz using an optically-pumped molecular laser as the source. The lasing gas was $C^{13}H_3F$ excited by the P32 line of a $CO_2$ laser. Details of this laser system have been previously reported (2).

The double-beam interferometer which was used for the measurement of indicies is shown in Figure 1. A number of "quasi-optical" components were combined to make this a rather unique instrument. $BS_1$ is a wire-mesh reflector which transmits a small fraction of the laser output to detector $D_1$ for power-monitoring purposes. The dielectric prism coupler (DPC) is a variable coupler which is based upon frustrated total internal reflection at the interface between two prisms. PS is a mechanical phase shifter and WG indicates the location of oversized dielectric waveguides which control diffraction spreading of the beam and maintain good mode quality. $BS_2$ is a mylar-film beam combiner and the final detector $D_2$ is a Golay cell. A He - Ne laser is boresighted to the beam for alignment purposes. S denotes the position of the sample which is mounted on a rock and pinion drive to permit reproducible insertion and removal.

A refractive index measurement was accomplished by first adjusting the phase shifter with the sample out to obtain a null at detector $D_2$, then inserting the sample and readjusting the phase shifter to return to a null condition. Letting L be the net path length change produced by the phase shifter, the index was calculated using the equation

$$n = 1 + \frac{N\lambda_o + L}{d} + \frac{\delta\lambda_o}{2\pi d}$$

Here $\lambda_o$ is the vacuum wavelength, d the sample thickness, N the integral number of wavelengths within the sample and $\delta$ a small correction

for multiple reflections in the sample (3). N can be computed if an approximate value of the index is available, otherwise, measurements on at least two different sample thicknesses are needed to determine N.

Measurement of the sample transmission for the purpose of determining the absorption coefficient was accomplished with minor modification of the instrument described above. The beam combiner ($BS_2$), Golay cell detector ($D_2$) and alignment laser (AL) are removed. This gives access to the two beams from the DPC. Two identical pyroelectric detectors were then placed to intercept these two beams. Their outputs were fed to a ratiometer which displays the ratio of the power in the sample beam to that in the other beam, which now serves as a reference. The first state of this measurement was to adjust the DPC to produce a reading of one on the ratiometer, with the sample removed. The sample was then inserted and the resulting ratio read as the fractional transmission of the sample.

For the case of radiation normally incident on a plane, parallel plate of thickness d, the transmission is related to the absorption coefficient $\alpha$ and the refractive index n by (4, 5)

$$T = \frac{\rho(1 - R)^2/R}{1 + \rho^2 - 2\rho\cos(2\varphi)} \text{ where}$$

$$R = \frac{(n - 1)^2}{(n + 1)^2}, \quad \rho = R\exp(-\alpha d), \quad \varphi = \frac{2\pi nd}{\lambda_o}$$

The first equation can be solved for the parameter $\rho$ and the absorption coefficient is then given by

$$\alpha = \frac{-\ln(\rho/R)}{d}$$

Computation of $\alpha$ from the measured transmission in this manner clearly requires an accurate knowledge of the sample's index and thickness.

## III. Discussion and Results

The values of refractive index and absorption coefficient which we have obtained for a number of materials are presented in Table 1. Where available, the results obtained by other investigators are shown for comparison. The calculated loss tangent is also tabulated for convenience.

All samples were in the form of discs with diameters between 25 and 50 mm and smoothly polished surfaces. Measurements were made on the central 19 mm of the disc. The significant sources of error in the index measurements are the uncertainty in the sample thickness and the uncertainty in the path-length change of the phase shifter. The errors quoted in Table 1 are standard errors. Representative standard errors for sample thickness and path length change are .002 mm and .005 mm respectively. The resulting uncertainties in our index values are typically 0.02% or less.

The error in the absorption coefficient depends not only upon the uncertainty in the measured transmission, but also upon the uncertainties in the sample's index and thickness. This computation is rather involved so a computer program was used to obtain the quoted estimates of error in the absorption coefficient. The resulting error estimates correspond to about 10% or less for the samples reported here.

### Quartz

Measurements were made on a sample of x-cut synthetic crystal quartz (6) oriented crystallographically within 2 minutes of arc, with a thickness of 7.991 ± 0.0003 mm. It has a very low-loss at this frequency and is readily available in pure form with high crystal quality. Our measurements of the ordinary and extraordinary indices at 245 GHz are 2.1059 and 2.1551, respectively. That is, the birefringence for the

crystal quartz amounts to a value of 0.0492. These values of the indices are in good agreement of the recently published values of 2.106 and 2.154 (7) respectively for the same sample. The uncertainty in our absorption measurement is high for such a low loss sample and we could only put an upper bound of .05 $cm^{-1}$ on this value. Use of a thicher sample can improve the level of confidence in our absorption measurement and such a measurement is being undertaken in this laboratory.

Fused Silica

The sample used in a silica glass produced by Thermal American Fused Quartz and designated as Spectrosil WF. It is distinguished by a very low water content (less than 10 ppm -OH). Our values for index and absorption coefficient are 1.9516 and .08 $cm^{-1}$, respectively. These are in good agreement with the results of Afsar and Button on a similar sample (9). It is noteworthy that this water-free glass has an absorption coefficient which is less than one-half that of a typical fused silica (Dynasil 4000) which was previously measured in this laboratory (2).

Silicon

The single-crystal silicon sample was obtained from General Diode Corporation. It is oriented with the (111) plane in the plane of the disc and slightly doped with boron (0.18 ppb) to achieve a resistivity greater than 1500 ohm-cm. Our values for the index and absorption coefficient are 3.4181 and .13 $cm^{-1}$, respectively and are in excellent agreement with those reported by Afsar and Button (9).

Beryllia

The sample used was K-150 material of the National Beryllia Corporation and was 99.5 percent chemically pure. This isopressed white disc had on the average, 22-μm grain size and a density of 2.90 $gm/cm^3$.

Values for index of refraction and absorption coefficient were 2.6126 and 0.100 $cm^{-1}$ respectively. Our index value agrees well with a recently published value of 2.60846 (44) for a similar sample with grains between 18 and 25 μm and density of 2.9086 (5) $g/cm^3$ (8). Value for absorption coefficient is also in good agreement with this published value of 0.12. However, our α-value is about one-half of that reported by Afsar and Button (9) at 245 GHz for their hot pressed beryllia. Such a difference may well be attributed to the variation in the ceramic manufacturing process.

Boron Nitride

The Boron nitride (grade HP) sample was a produce of Carborandum (10). It is an ceramic material with a chemical analysis of 42 percent boron, 53.5 percent nitrogen, and 1.5 - 2.5 percent oxygen. Density of this sample was 1.90 $gm/cm^3$. It is hot pressed and the pressing axis is perpendicular to the disc surface. Our results for index and absorption coefficient are 2.0727 and 0.068 respectively. As far as we know, there are no published data for this material at NMMW. This material which can withstand temperatures up to 3000°C can be machined to close tolerances. In terms of loss and index value, it appears to be superior to the finest dense alumina ceramics.

Nickel-Ferrite

This sample is a commercial product of Trans-Tech, Inc. designated as Type 2-111 (11). It is a ceramic material with an average grain size in the 10-15 micron range. The saturation magnetization and resistivity of this sample were given by the supplier as 5200 gauss and $8.96 \times 10^7$ ohm-cm, respectively. We obtained values for the index and absorption coefficient of 3.7298 and 0.33 $cm^{-1}$, respectively. This index value is

in good agreement with a recently reported result which was obtained with a laser technique similar to ours (7). However, our value for the absorption coefficient is approximately one-half that which was obtained by the same investigators with an FTS technique (7).

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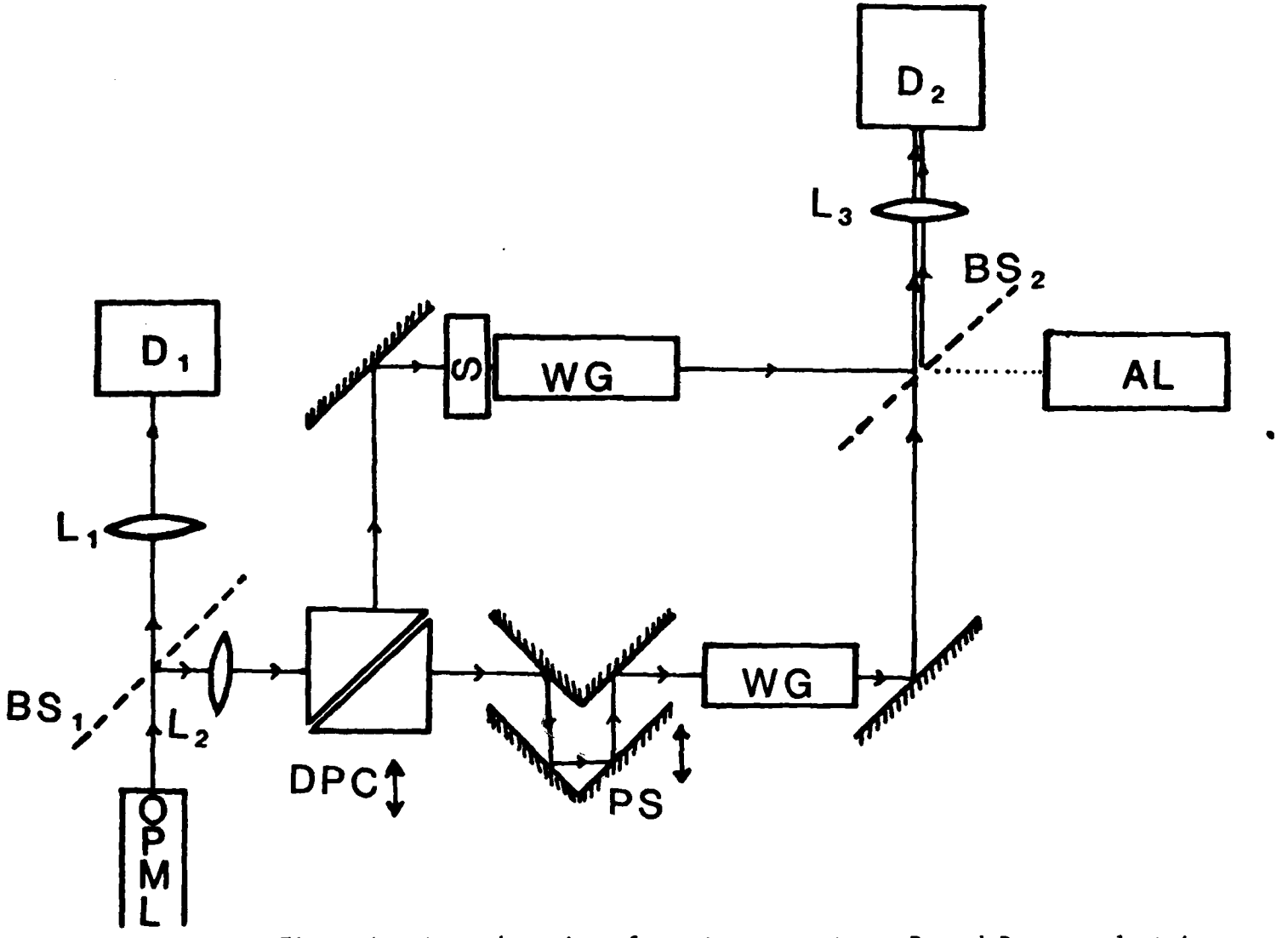


Figure 1. A two-beam interferometer apparatus. $D_1$ and $D_2$, pyroelectric detectors (Laser Precision Rkp-545): $L_1$, L, and $L_3$, TPx lens; $BS_1$, wire-mesh beam splitter; $BS_2$, mylar-film beam splitter; DPC, double-prism coupler; PS, phase shifter; S, sample holder; WG, dielectric waveguides; and AL, He-Ne alignment laser.

TABLE 1

EXPERIMENTAL RESULTS

| Material | This Work at 245 GHz | | | | Literature | |
|---|---|---|---|---|---|---|
| | Thickness (mm) | n | $\alpha(cm^{-1})$ | Loss Tangent ($10^{-4}$) | n | $\alpha(cm^{-1})$ |
| Quartz-O | 7.991 | 2.1059 ± .0004 | <0.05 | | 2.106 (Ref. 7) | |
| Quartz-E | 7.991 | 2.1551 ± .0004 | <0.05 | | 2.154 (Ref. 7) | |
| Fused Silica | 20.007 | 1.9516 ± .0002 | .081 ± .003 | 7.97 | 1.95117 (Ref. 9) | 0.07 (Ref. 9) |
| Silicon | 10.1829 | 3.4181 ± .0007 | .134 ± .015 | 7.62 | 3.41805 (Ref. 9) | .1303 (Ref. 9) |
| Beryllia | 20.6286 | 2.6126 ± .0002 | .100 ± .015 | 7.44 | 2.60846(44) (Ref. 8) | .125 (Ref. 8) |
| Boron Nitride | 13.5712 | 2.0727 ± .0003 | .068 ± .003 | 6.38 | 2.05 at 94 GHz (Ref. 12) | |
| Nickel Ferrite | 12.7051 | 3.7298 ± .0007 | .334 ± .025 | 17.4 | 3.73 ± .043 (Ref. 7) | .62 (Ref. 7) |

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